Standard Operating Procedure for Sampling of Particulate-Phase Organic Carbon, Nitrogen, and Phosphorous in Great Lakes Waters

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1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure describes the sampling of Great Lakes Waters for particulate-phase organic carbon (POC), nitrogen (PN) and phosphorous (PP).

1.2 Samples of lake water are collected and passed through a 0.7 µm pore-size glass fiber filter for POC, 0.45-µm Sartorius cellulose acetate filter for PN and 0.45-µm Sartorius cellulose acetate filter for PP. POC, PN and PP are operationally defined as the mass of organic carbon, nitrogen and phosphorous retained on the filter per unit volume of water that passes through the filter.

2.0 SAFETY AND WASTE HANDLING

2.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures and; 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.

2.2 All applicable safety and waste handling rules are to be followed. These include proper labeling and disposal of chemical wastes. Over-board discharges of chemical wastes are forbidden.

2.3 During sampling, caution, common sense, and good judgment should dictate appropriate safety gear to be worn in any given situation on deck. Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet; however, if in doubt, please ask the Chemical Hygiene Officer.

2.4 Collecting samples in cold weather, especially around cold water bodies, carries the risk of hypothermia and frostbite. Sampling team members should wear adequate clothing for protection in cold weather. For specific information regarding sampling during cold conditions, please refer to the US EPA GLNPO *Standard Operating Procedures for Winter Operations* (December 1994, or as amended).

2.5 Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.

2.6 Work vests must be worn while working on the fantail and rosette deck.

3.0 SUMMARY OF PROCEDURE

3.1 Great Lakes water samples are collected at the Rosette sampler.

3.2 The water is then filtered under vacuum through the filters in an all-glass filtration apparatus.

3.3 The POC samples are acidified during the filtration to remove inorganic carbonates. The particles are retained on the filter and frozen at -10°C until analysis.

4.0 DESCRIPTION OF APPARATUS

4.1 Water samples (typically 1-4 liters for open-lake locations) are collected from Rosette sampler.

4.,2 Cellulose acetate and ashed glass fiber filters are supported in commercially available all-glass 350-mL vacuum filtration apparatus. Samples are filtered under the vacuum between 5-10 inches of Hg. Tygon tubing (3/8" ID) is used to connect the filtration flasks to a ship vacuum. The equipment needed is listed in Table 1.

5.0 PREPARATION OF FILTERS AND REAGENTS

5.1 Preparation of Filters for POC

5.1.1 Filter preparation should take place as close to the start of the survey as possible.

5.1.2 Filters are to be handled only with stainless steel forceps. Filters that are mishandled after the ashing procedure (5.1.4) should be discarded.

5.1.3 47-mm diameter GF/F filters (0.7-µm pore-size) are placed individually in aluminum foil envelopes, dull side of foil facing inward, with three sides folded closed. The fourth side is left open to allow gases to escape from the envelope during ashing.

5.1.4 The filters are stacked in a muffle furnace and ashed for four hours at 450°C.

5.1.5 Upon removal from the muffle furnace, the envelopes are sealed on the fourth side.

5.1.6 Fifty envelopes containing individual filters are placed into a Ziploc bag and the bag is labeled with the date and initials of the analyst who prepared the filters.

5.2 Preparation of Reagents

A solution of 0.2 N HCl is prepared by transferring 17 mL of concentrated HCl (16.1 N) to a 1000-mL volumetric flask and diluting to the mark with organic-free, distilled, reagent water. Transfer the solution to a Teflon wash bottle.

6.0 FILTRATION PROCEDURE

6.1 Using stainless steel forceps, place one filter onto the glass support of the sampling apparatus. Place the funnel on top of the filter and secure with the clamp. Label the Great Lake name, station number, sampling depth, and date onto the aluminum foil envelope for POC samples and onto Petri dishes for PN and PP.

6.2 Measure the volume of lake water to be filtered in a graduated cylinder, or mark all sampling bottles at the 1 liter level by using the graduated cylinder. Prior to filling, rinse the bottles, or cylinders, twice with approximately 100 mL, of lake water.

6.3 Pour the measured volume of lake water into the glass filtration funnel. Turn on the vacuum. Maintain the vacuum between 5-10 inches of Hg and pour lake water into the funnel until sufficient material has been collected (see section 6.7). In case of filtration for POC, just before the last portion of the lake water has been filtered, squirt some volume of 0.2 N HCl solution into the funnel, enough to cover the filter surface (approximately 5 mL).

6.4 The volume of lake water required to produce a reliable POC, PN and PP measurement (i.e., an amount of material that is within the analytical instrument's linear range) will vary with lake station location, depth, and time of year. For open-lake, oligotrophic conditions, typically 1-4 liters will provide enough material. For near-shore locations, or meso-eutrophic and eutrophic conditions, lake water volumes in the range of 200-500 mL are typical. A filter that becomes visibly loaded with particles and a flow of water through the filter that drops significantly, are evidence that sufficient particulate material has been collected.

6.5 After the lake water has been filtered, rinse the sides of the funnel with approximately 20 mL of reagent water and filter this rinse. Turn off the vacuum.

6.6 Remove the funnel. Using stainless steel forceps, fold the filter in half and place back it into the labeled aluminum foil envelope or in pre-labeled Petri dish for PN and PP. Foil envelopes and Petri dishes with filters should be stored at -10°C. Record the Great Lake name, station number, sampling depth, volume filtered, analyst, date, and time of day on the Sampling Log Sheet.

6.7 Empty the remaining filtrate from the filtration flask.

6.8 Rinse the filtration funnel, filtration flask, and the container(s) with reagent water.

6.9 Re-assemble the filtration apparatus.

6.10 Place aluminum foil covers over the filtration funnels.

7.0 QUALITY CONTROL

7.1 Laboratory duplicates are analyzed approximately every fourth sample for each of the parameters.

7.2 Field reagent blanks are analyzed approximately every fourth sample for each of the parameters.

Table 1: List of Filtration Equipment

Equipment	Source or Equivalent	Quantity
Vacuum and pressure regulator	Cole-Parmer H-07061-30	2
350-mL All-glass filtration apparatus	Nucleopore	3
Plastic bottle (1000-mL)	Fisher 02-893D	4
Teflon wash bottle (500-mL)	Fisher 03-49-12E	2
Teflon bottle (1000-mL)	Fisher 02-924-15G	2
Forceps	Fisher 10-317-5	2
Tygon tubing (3/8"ID)	Fisher 14-169-2C	1

Miscellaneous (some quantities depend on number of samples)

- 47-mm GF/F filters (0.7-µm pore-size) Whatman 1825-47
- 47-mm Sartorius cellulose acetate filters
- Petri dishes
- Ziploc freezer bags (1-gallon)
- Aluminum foil
- Permanent markers